Aug 6th, 9:00 AM - 12:00 PM

Raman studies of 1,3,5,7 Cyclooctatetraene at high pressure

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Repository Citation

Galley, Martin; Romano, Ed; Tkachev, Sergey; and Pravica, Michael, "Raman studies of 1,3,5,7 Cyclooctatetraene at high pressure" (2008). *Undergraduate Research Opportunities Program (UROP)*. 27.  
https://digitalscholarship.unlv.edu/cs_urop/2008/aug6/27
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We performed Raman spectroscopic studies of 1,3,5,7-cyclooctatetraene at elevated pressures up to 10 GPa with the aim of examining possible planarization of the molecule and further studying two prior-discovered phases of the solid with pressure. The Raman excitation source was a Krypton-ion laser operating at 674.1 nm (give wavelength).

1,3,5,7 Cyclooctatetraene has an octagonal formation however it is not aromatic or anti-aromatic (not a subject to the 4n+2 Hückel's rule) [1]. As a result, its adopts a somewhat reactive tub shape. Upon the addition or removal of one to two electrons under ambient conditions, the molecule planarizes and becomes aromatic [3-5]. Our goal was to determine if we can aromatize the COT molecule mechanically or via electron transfer at elevated pressure.
Raman Studies of 1,3,5,7 Cyclooctatetraene at High Pressure
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Introduction
1,3,5,7 Cyclooctatetraene has an octagonal formation however it is not aromatic or anti-aromatic (not a subject to the 4n+2 Hückel’s rule) [1]. As a result, its adopts a somewhat reactive tub shape. Upon the addition or removal of one to two electrons under ambient conditions, the molecule planarizes and becomes aromatic [3-5]. Our goal was to determine if we can aromatize the COT molecule mechanically or via electron transfer at elevated pressure.

Abstract
We performed Raman spectroscopic studies of 1,3,5,7 cyclooctatetraene (COT) at elevated pressures, up to 11 GPa using a diamond anvil cell (DAC), with the aim of confirming two prior-discovered phases of the solid under pressure [6]. After reaching 11 GPa the pressure was decreased to ascertain the reversibility of high pressure effects on COT. The Raman excitation source was a Krypton-ion laser operating at 647.1 nm and the spectrometer used was a Triax 555B with an attached CCD detector.

Compression
Decompression

Procedure
We used a Merrill-Bassett diamond anvil cell with 500 micron culet and then pre-inserted a lithium gasket to about 50 microns thick. We drilled a 210 micron hole through the gasket and then placed a 8-10 micron ruby chip as well as the liquid cyclooctatetraene into the gasket hole. Pressure was then increased until the first sign of the COT sample traversing a phase change towards a solid is reached. We then acquires the Raman spectra using a Krypton-ion laser with wavelength of 647.1 nm, and stored data over four different Raman wavenumber ranges (200-800; 000-1350; 1350-2000; 2780-3300 cm\(^{-1}\)). This was performed for each pressure point until the sample began to show signs of reaction (possibly polymerization) around 11 GPa. Pressure was then decreased and Raman spectra were taken over the same wavenumber regions. The acquired data are displayed in the figures on the right.

Conclusion
We performed Raman spectrometry on a COT sample as a function of pressure at ambient temperature. Raman spectra from 20 different pressure points were recorded from 0.05 GPa to 1.17 GPa. This study revealed two novel phase transitions which were prior observed in this group using a 514.5-nm laser (Argon ion) but has better determined the pressure at which the phase transitions occur [6]. The sample was also studied in decompression to ascertain reversibility/survivability of the sample with pressure. In leaving a sample under 11 GPa of pressure for over one week, partial reaction and/or polymerization had occurred. When COT has entered this amorphous state it becomes more sensitive to the effects of the laser, as black marks developed where the laser rested. These tests were inconclusive in determining if COT had aromatized while at high pressure. Further study will be needed.

Acknowledgments
We thank Michael Hake for aid in the measurements and experimentation process. Support from the Research Experience for Undergraduates (REU) program of the National Science Foundation is gratefully acknowledged under grant DMR-0552999.

We gratefully acknowledge support from the U.S. Department of Energy Cooperative Agreement No. DE-FC03-01NA64249 with the U.S. Department of Energy, and also wish to thank the NASA/Nebraska Space Grant for greatly supporting this work.

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